HIGH TEMPERATURE THERMOCOUPLE RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 5
Period 1 October 1963 to 1 November 1963
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
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Huntsville, Alabama

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ABSTRACT

This report covers the period 1 October 1963 to 1 November 1963, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C range, under adverse conditions of oxidation, erosion, vibration and shock.

The primary objectives of the program are to advance the state-of-the-art of high temperature thermometry, and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Three prototype gauges were delivered, as shceduled, on 17 October 1963 for test and evaluation. Calibration curves for these items indicated that their output, although parallelling temperature - emf curves taken during previous calibrations, was shifted to a higher emf value for the same temperature. After review of the characteristics of various types of compensated lead wires, it is believed that the shift is caused by a spurious attributable to the particular type of lead wire used in the prototype assemblies. Preparations were made for fabrication of test articles and test setups for tests in the 3000°F to 5000°F temperature range.

SECTION I

SUMMARY

1.0 Period Covered

This report covers the period 1 October 1963 to 1 November 1963.

1.1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion, oxidations and high stress levels for useful periods of time. Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle.

To accomplish the above objectives, the Contractor shall consider and explore specific R&D efforts as follows:

a. Development of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.

b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.

c. Determine the best combination of compensated lead wires for use with the immersion type probes.

d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.

1.1 Statement of Work Cont....

e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output.

f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

Accomplished during the current reporting period were:

a. Parts Fabrication

Three prototype gauges were fabricated, and were delivered to M-ASTR-I on 17 October 1963, as scheduled.

b. Conferences

An ACL representative met with M-ASTR-I personnel for discussions of aspects of the end use requirements of the gauges to be produced under this project. It was agreed in this meeting that ACL would request, in writing, definition and clarification of various physical, thermal, and electrical parameters necessary to completion of the second four month period, under the contract. These requests are set forth in Section III of this report. A meeting was also held with M-P&C-MPA to review the contract and required submissions thereto.

Further correspondence has also been exchanged with the Government Property Administrator, Los Angeles, California to assure that ACL procedures for handling Government Property, under the contract, agree with all currently applicable documentation.

1.2 Progress Cont....

c. Test Oven

During a test intended to expose a test gauge to temperatures in the 3000°F to 5000°F range, under oxidizing conditions, the outer structure of the test duct exploded when moisture entrained in the insulation flashed over into steam. No injuries to personnel were sustained, nor was there damage to equipment, other than the test duct. As a consequence, calibrations and oxidation tests will be delayed until the next reporting period. Work is proceeding on repairing and improving the duct, to prevent a recurrence of this incident.

d. An analysis of the form drag and shock drag of the Type 4735 gauges, as seen in the prototypes has been started.

e. Lead Wire

Evaluation samples of compensated lead wire have been ordered from Minneapolis-Honeywell, Englehard Industries, and Tuttle & Kifft. One test gauge has been fitted with two sets of leads of different types. Provision for switching to determine differences in output has been made.

f. Analysis of Prototype Evaluation Tests

It was not possible to perform evaluation tests at M-ASTR-I at the time of delivery of the prototypes. This analysis will, therefore, be performed when test results and associated data are available.

g. Ceramic Search

The search for high temperature insulators was continued. As yet, no positive results have developed. All manufacturers and research facilities contacted, thus far, have reported no such material is presently available, although intensive research is being conducted in this field.

1.2 Progress Cont....

h. Oxidation Resistant Coatings

As stated in Paragraph "f" above, no prototype test results were available as of the date of this report. Tabulations of oxidation tests are presented in Section III of this report. ACL is setting up for oxidation tests at higher temperatures, and will report results in the next report.

i. SRI Report, Type 4734 Gauges

As previously reported, Southern Research Institute conducted an independent evaluation of a number of ACL Type 4734 gauges, which are similar to the ACL Type 4735 gauges. A copy of their final report* was made available to ACL during the current reporting period. Results of their tests were analyzed, and were found to agree essentially with evaluations conducted previously by ACL.

*Final Report on Task Order 1, Contract NAS 8-5196
Southern Research Institute, Birmingham, Alabama

SECTION II

PAST PROGRESS

2.0 General

Previous effort was reported in ACL Progress Reports T-1097-1 through T-1097-4.

2.1 Prototype Design and Development

As was previously reported, objectives for the first prototypes have been limited to the 4000°F - 4500°F range in the interest of accumulating test data for analysis, the results to be utilized in future design.

A design approach for the prototype gauges was selected, and drawings prepared, detailing means of fabrication and assembly.

Investigations made into fabrication techniques involved in working vapor deposited Tungsten, resulted in improved material handling techniques.

Shock and vibration tests, performed on a prototype mock-up, resulted in a conclusion that the sheath material was intrinsically capable of withstanding the specified shock and vibration requirements.

Samples of various types of compensation lead wires were ordered for test and evaluation. All samples were not received in time for test.

An evaluation of the SRI calibration tests for ACL Type 4734 gauges was made, resulting in a conclusion that an optimum immersion depth might be in the order of 1-1/2 inches in an isothermal region.

Preparations were made for examination of the two Type 4734 gauges tested by N.A.S.A.

2.1 Prototype Design and Development Cont....

A test of a "no-insulation" approach was started, but was aborted due to a failure in the test oven.

Three prototype gauges were scheduled for delivery to M-ASTR-I, on 17 October 1963, for test and evaluation. Calibrations of this type of gauge indicated a shift in emf output to a higher value than that shown in previous calibrations. The shift was believed due to a spurious emf contributed by the "compensated" lead wires. The curves, however, parallel the curves taken by Southern Research Institute, as well as those predicted by ACL.

SECTION III

CURRENT PROGRESS

3.0 General

Three prototype gauges, ACL Type 4735, were delivered to M-ASTR-I on 17 October 1963, as scheduled.

3.1 Progress

3.1.1 Prototype Gauges

The three prototype gauges delivered during the current reporting period were assigned serial numbers 002, 003, 004 for purposes of identification. All of these prototypes were fabricated in the same manner, to permit correlation of any data resulting from test and evaluation performed by M-ASTR-I.

3.1.2 Construction Details

The prototype gauges were assembled in general conformance with the fabrication details described in Report T-1097-4. Deviations to the methods and materials described in that report are as follows:

a. Oxidation Protective Coating

When the probes were received from the coating process, the elemental silicon overlay was found to have lost adherence to the siliconized sheath in some areas. Where possible, the loosened silicon was removed. The sheaths were not mechanically worked to entirely remove the elemental silicon, to minimize the possibility of inadvertently providing nuclei for grain growth. (See Report No. T-1097-3, Para. 3.1.2.1) The D-65 material was not applied to the base of the probe, near the union because of

a. Oxidation Protective Coating Cont....

the possibility of the introduction of errors in emf output due to the creation of a thermal gradient (tip to base) different than that seen in the gauges used in calibrations. Future calibrations of gauges so treated will be made to determine the effect of the D-65 material.

3.1.3 <u>Recapitulation, Oxidation Effects Type 4735 Gauge Ser. No. 001</u>

All tests performed by ACL up to 3000°F, under oxidizing conditions, had not been completed during the last reporting period. A recapitulation of these tests, including a run to destruction, is presented in Table I. A further breakdown shows the following: 4 hr. 37 min. running time up to 2000°F, 25 min. additional running time from 2000°F to 3000°F.

The sheath material in the Serial No. 001 probe was elemental Tungsten, .030 inches wall thickness. No protective coating was employed. The mock-up used in vibration tests, fabricated of the same material, but with a wall thickness of .010 inch (incorrectly reported in Report No. T-1097-3 as .020 inch) was then subjected to a run to destruction in the same test setup used with the 001 probe. A recap is presented in Table II.

TABLE I

RUNNING TIME AT TEMPERATURE

Type 4735 Gauge, Serial Number 001

Run No.	Run Time Per Run Hrs.	Run Time Per Run Min.	Run Temp °F	Total Run Time Hrs.	Total Run Time Min.	Remarks
1	1	10	1300°F to 2200°F	1	10	Atmosphere Argon Slight oxidation
1-1		30	1300°F to 2200°F	1	40	Argon Slight oxidation
2		30		2	10	Air, blue and purple oxides
3	2	27	1300°F to 2100°F	4	37	50% Argon 50% Air, blue and purple oxides more pronounced
3-1		5	2768°F to 2984°F	4	42	Oxy-Acetylene Burner, yellow and green oxides started to form
3-2		7	3029°F to 3182	4	49	Yellow oxides more pronounced
3-3		6	2102°F to 3182°F	4	55	Yellow oxide formation, erosion apparent on 1st 1/2-inch of tip. Output good
3-4		5	2966°F to 3029°F	5	00	Yellow oxide formation, probe still functions.
3-5		2	2966°F to 3029°F	5	07	Heavy oxidation, output dropped off at 2 min. Probe failed

TABLE II

RUNNING TIME AT TEMPERATURE

MOCK-UP GAUGE

Run No.	Run Time Per Run Min.	Run Time Per Run Sec.	Run Temp °F	Total Run Time Min.	Total Run Time Sec.	Remarks
1	2	00	2500°F to 3000°F	2	00	Green and yellow oxides forming
2	4	00	2600°F to 3000°F	6	00	Heavy formation of green and yellow oxides
3	2	30	2600°F to 3000°F	8	30	Heavy yellow oxides formed, probe failed

3.1.4 No-Insulation Approach

In the early attempts to run the no-insulation tests, a gross error in concept invalidated the first runs. In these tests, the simulated thermocouple was located in an isothermal zone. No errors in output, as compared with a standard thermocouple probe were noted. At first inspection, no notice was taken of the validity of the setup itself. Upon re-examination it was apparent that no useful information could come from the setup as made. With the entire test block and the thermoelectric elements at the same temperature when the oven was stabilized, there would be no temperature differential existing at any point in the immersed portion of the system.

The major objective of the test was to find whether a probe could be constructed without electrical insulation such that current leakage between conductors of the pair in the absence of an insulator would not deteriorate the output from the junction to the extent that the probe would be rendered useless. With the system in an isothermal zone, and at equilibrium, all parts of the system would be at the same temperature with the exception of a small differential due to thermal conduction along the lead wires (which were of the same material as the thermocouple). If current leakage between the legs of the pair occurred, in the isothermal zone, the effect would be identical to that occurring in a set of thermocouples hooked up in parallel. That is, the net effect of the leakage would be to average the outputs of a large number of parallel junctions, all at the same temperature. Therefore, the setup is being revised to include the presence of a suitable temperature differential.

3.1.5 Test Duct

As reported in the last monthly progress report, it was planned to conduct calibrations in the high temperature duct at temperatures in the 4000°F range. The setup was made and the test started. When, however, the burner was ignited, moisture entrained in the duct insulation flashed over to steam, and an explosion followed. The duct is being rebuilt to avoid such a recurrence. There was neither injury to personnel, nor damage to other equipment.

3.1.6 Oxide Insulators

As evidenced in both the Southern Research Institute report and the ACL tests, if presently available electrical insulators are used in a high temperature probe, the upper operating temperature is limited by the characteristic behavior of the insulator. That is: the insulator becomes conductive at some elevated temperature.

There is wide disagreement between leading authorities as to the exact mechanism of the change in the ceramic from insulator to conductor. At present, the consensus is preponderantly in favor of Beryllia as the most effective of the oxides, and probably the best of the high temperature insulators. In meetings with research personnel at Los Alamos, the merits of Beryllia, Magnesia, Thoria, and Hafnia were thoroughly discussed. In their experience, an upper limit of 4200°F to 4500°F is attainable with Beryllia in applications of the type under consideration in this project. Other sources* report the presence of even small amounts of other materials effects a significant change in the phase change temperature of the internal structure of the material.

The maximum temperature of effective use, without degredation of insulation characteristic is greater by consensus, with Beryllia than with the other ceramics.

3.1.7 Gas Dielectrics

One area of approach, although inferrenced as a possibility in previous reports, had not been given serious attention until current evidence indicated that availability of a true high temperature insulator is unlikely within the time imposed by this contract. In investigating the electrical insulation characteristics of other materials, it was found that some materials, undergoing a change of state at 600° to 700°C, become a gas with very high dielectric strength. One such material is Sulfur Hexafluoride. It is planned, therefore, to further investigate such materials and their possible application to the no-insulation approach discussed elsewhere in this report.

*Guide to Refractory Ceramics, Materials in Design Engineering Vol. 58, No. 1, PP 86 - 96 July 1963

RTD Technology Briefs, Vol. 1, No. 10, PP 1 - 6 Oct. 1963 Hq, Research & Technology Div'n. Bolling AFB. Wash. 25. D.C.

3.1.8 Requests for Information

During the visit by ACL personnel on 17 October 1963, certain requests for information regarding the gauges to be developed in this program were discussed with M-ASTR-I personnel. Such information is related principally to the ultimate conditions of use, and possible mounting and orientation configurations. These are listed below.

a. A determination whether a means of mounting, other than through a 7/16"-20NF mounting boss could be employed. The physical size of the probe limits the designer to small spacing internal to the probe, possibly imposing restrictions on performance at maximum operating temperatures.

b. A description of the desired orientation of the gauges, relative to flow. At present, it is not known by ACL whether flow will impinge upon the gauge on its side or on its tip. (As seen in previous rocket engine tests) Thus, the designer is limited to a symmetrical body of revolution (in cross section) in order to effect a compromise between all conditions of flow. It is realized that a multi-purpose gauge would be desirable, however, present thinking indicates that the multi-purpose gauge would not permit the most desirable geometry for a given condition of flow.

c. The time for use at the maximum anticipated temperature of use, and an estimate of the time-temperature profile. This information, together with that described in b. above, is of great importance to the designer in that the loadings on the immersed portion of the probe are influenced by the velocity and mach number of the medium. During the temperature rise, from light-off to steady state conditions, the mach number could change from sub-sonic, to supersonic, in a very short time. The character of the aerodynamic loading of the probe, could thus change very rapidly, and the imposition of shock loading could influence the geometry to a great degree.

d. The length of lead wire required in the installation, the required insulation resistance in the lead wire, and any shielding requirements.

e. A desired immersion depth, in terms of distance from the inside wall of the duct, chamber, etc., to the tip of the gauge.

3.1.8 Requests for Information Cont....

f. A description of the wall into which the gauge will mount, including thickness, materials, and a cross section sketch, if available.

g. An estimate of the velocity of the medium in which the gauge is intended to run.

h. A description of the medium in terms of its chemical constituents, density, and ratio of specific heats.

3.1.9 Analysis of SRI Final Report

A copy of the Southern Research Institute Final Report, covering calibrations and oxidation tests of ACL Type 4734 gauges, was obtained during the current reporting period. There is basic agreement on all phases of their observations and measurements except as follows:

a. In the SRI summary it was indicated that errors in measurement, or faulty techniques were responsible for differences in emf vs temperature between the predicted curve and the SRI curves. The ACL curve was first calculated, then compared with the Englehard curve for W-W26Re. Good agreement was obtained. Spot checks were then taken, in an isothermal zone, and when agreement between the calculated curve, the Englehard curve, and the spot checks was observed, the curve was drawn, based on the Englehard curve, with a reference junction temperature of 0°C. From the SRI curves, wherein differences in output, at the same temperature, for different immersion depths were seen, it can be inferred that conduction losses were largely responsible for the deviations from the ACL curve. The original ACL spot checks were run at an immersion depth of at least 25 diameters, whereas the SRI calibrations were taken at 5-1/4 to 6 diameters. Inspection of the SRI curves shows that the output for a discrete temperature moves toward the ACL curve as the immersion depth is increased. In a previous ACL progress report, the deviation was plotted as a function of immersion depth. The resulting curve became asymptotic as the immersion depth was increased. This satisfies both the first law of thermodynamics and the literature regarding "end" or conduction losses.

3.1.9 Analysis of SRI Final Report Cont....

b. Spurious emf's due to the lead wire are mentioned in the SRI report as possibly contributing to differences between the SRI calibrations and the ACL calibrations. This observation is valid. However, based on both the SRI calibrations, and later ACL calibrations, as well as the discussion in a. above, the errors due to any spurious emf contributed by the compensated lead wire must have been quite small. Of greater interest is the near certainty that a much greater spurious emf exists with different types of lead wire. This effect is believed to have been responsible for the appreciable differences between the SRI curves and the recent ACL curves. In these cases the essential difference between the Type 4734 gauges tested by SRI, which employed a copper (+) and copper-nickel (-) lead wire, and the Type 4735 gauges, which employed two different copper-nickel alloys, calibrated by ACL was in the compensated lead wire. Both employed the same type of junction, (W-W26Re) therefore, the emf for the same temperature, should have been the same. The ACL tests were made in two ways; with the sheath assembly alone (with lead wires attached) and with the sheath assembly mounted in the body. No observable differences were noted in these tests, thus further reinforcing the belief that the spurious emf, if present, was due to the copper-nickel alloy positive lead wire.

c. In the burner calibration tests, ACL agrees with the method used in the tests, except for contact of the sheath with the graphite cylinder. This method, although normally yielding good results thermally and with respect to emissivity, can contribute to an accelerated deterioration of the sheath due to the possible reaction between the Tungsten sheath and the graphite. This has been observed in previous ACL tests. The rise in observed temperature from the first to the last cycle can be explained if the graphite tube deteriorated enough, during a given cycle, to allow an increase in flame leakage. Great difficulty has been experienced by nearly all researchers in stabilizing temperatures during burner tests. Even small variations in burner pressure, flame path, or probe position can effect appreciable local variations of temperature. These are particularly evident at the higher temperatures, and are at times difficult to detect due to the time required to obtain a good brightness match in the optical pyrometer during a temperature change.

3.1.9 Analysis of SRI Final Report Cont....

d. In the SRI evaluation of divergence between the burner exposure and cavity calibration, it is interesting to note the very high degree of repeatibility of the Type 4734 gauge, on successive cycles, with points taken on the first through the fourth cycles being nearly coincident, and certainly falling within the degree of accuracy possible with the optical pyrometer under the conditions of test.

e. One significant aspect of the SRI tests, which was not discussed in the report, but which is worthy of mention is the absence of "flash" readings near the burner temperature (given as between 4760°F and 4870°F). If the flame ambient was in this range, it could be expected that flash readings could be seen, particularly when it is considered that the heat flux density was in the order of 600 BTU/FT^2/SEC. In previous tests such initial readings have been seen, quickly dropping off to some intermediate value in the gradient as the probe stabilized out at a temperature near the upper operating temperature of the Beryllia. It is possible, therefore, that the points in the burner flame, at which the readings were taken, were in a region of much lower temperature, and this fact was inadvertently omitted from the SRI report.

Despite disagreement with some details of the SRI report, and some discrepancies noted, ACL is in general agreement with the body of the report, and the conclusions.

SECTION IV

PROGRAM FOR NEXT INTERVAL

4.0 Objectives for the interval 1 November 1963 to 1 December 1963.

a. Continue calibrations and oxidation tests in the rebuilt high temperature duct.

b. Continue lead wire tests and compare results obtained from different types.

c. Analyse results of N.A.S.A. prototype evaluations, if available.

d. Continue search for insulators.

f. Start design of second generation gauges.

SECTION V

STATEMENT OF MAN HOURS

5.0 Hours by Category

Category	Previous Periods	Current Period	To Date
Engineering	376.50	65.5	442.00
Clerical	51.50	19.0	70.50
Fabrication	301.75	233.25	535.00
Consulting	-0-	5.5	5.50
Drafting	19.00	13.0	32.00